[Geochemical Analysis & Lab]

EXPERIMENT 5: Alkalinity Measurement by Gran Titration: When the Conductivity is below 100 S

**Preparation**:

* pH meter (calibrated), meter stand, probe holder
* Water sample 50mL (filtered)
* H2SO4 (95%), deionized water
* Magnetic stirrer, magnetic bar
* PE bottle (250mL, 50mL), beaker 100mL
* Micropipette (0.05mL, 1mL), pipette (10mL), volumetric pipette (50mL)
* Chemical balance

**Procedures:**

1. Preparation of 0.2N and 0.02N H2SO4
   1. Clean and dry a 250mL PE bottle
   2. Place the PE bottle on the chemical balance and tare (make the reading 0.0000)
   3. Add 1.313g of H2SO4 into the bottle (record the actual weight on Lab notebook)
   4. Move the bottle to the weighing pan of a top loading balance
   5. Add 123.687g of DIW to the bottle (record the actual weight on Lab notebook)
   6. Mark “0.2N H2SO4” with the group name and date on the bottle
   7. Clean and dry a 50mL PE bottle
   8. Place the PE bottle on the chemical balance and tare
   9. Transfer 10g of 0.2N H2SO4 from step *f* to the PE bottle (record the actual weight on Lab notebook)
   10. Add 90g of DIW to the bottle (record the actual weight on Lab notebook)
   11. Mark “0.02N H2SO4 “ with the group name and date on the bottle
2. Place a clean and dry 100mL beaker on a magnetic stirrer
3. Transfer 50mL sample to the beaker.
4. Immerse a Teflon-coated magnetic bar into the beaker and slowly turn on the stirrer. Control the stirrer speed not to make a vortex.
5. Turn on a calibrated pH-meter and immerse the pH probe into the sample. Record pH and T of the sample.
6. Add 0.05mL 0.2N H2SO4 to the sample. Wait until the reading stabilized and then record # of addition and pH.
7. Repeat step 6 until pH drops below 4.5.
8. Add 0.05mL 0.02N H2SO4 to the sample. Wait until the reading stabilized and then record # of addition and pH.
9. Repeat step 8 until pH drops below 3.5.
10. Plot F=(Vo + Vt)\*10-pH on the vertical axis vs. Vt on the horizontal axis, where Vo and Vt are the volumes of the sample and acid added, respectively. Vt is calculated by (10\*Vt1 +Vt2), where Vt1 and Vt2 is the volumes of 0.2N and 0.02N H2SO4 added.
11. Do a linear regression of the linear part. The slope of the regression line would be the actual normality of the acid Nt and the intercept of the line at the horizontal axis would be the equivalence volume Ve.
12. Calculate the alkalinity of the sample as below

Alkalinity = Nt\*Ve/Vo\*1000\*50 (mg CaCO3/L)

**Notes:**